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DISCHARGE CHARACTERISTICS OF LIAL/NAALCL SUB 4/FECL SUB 3 THERM--ETC(U)
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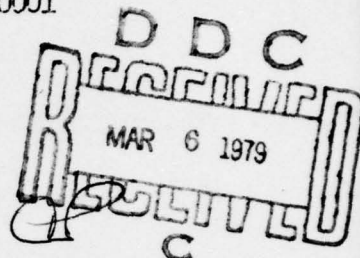
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FRANK J. SEILER RESEARCH LABORATORY

FJSRL TECHNICAL REPORT - 79-0001

FEBRUARY 1979



DISCHARGE CHARACTERISTICS OF
LiAl/WaAlCl₄/FeCl₃ THERMAL CELLS

CAPT ROBERT L. VAUGHN
LT COL LOWELL A. KING

PROJECT 2303

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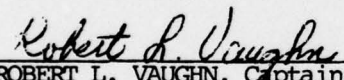
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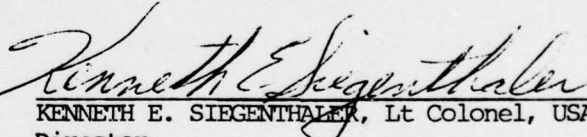
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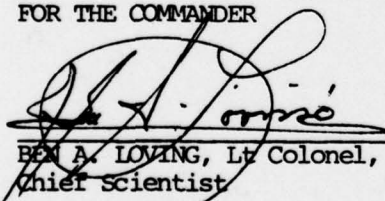
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250°C and 15 mA/cm² to 100 mA/cm². Iron(III) chloride cells were comparable to or superior to the molybdenum pentachloride and copper(II) chloride cells for these operating ranges.

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DISCHARGE CHARACTERISTICS OF
 $\text{LiAl}/\text{NaAlCl}_4/\text{FeCl}_3$ THERMAL CELLS

By

Capt Robert L. Vaughn
Lt Colonel Lowell A. King

TECHNICAL REPORT FJSRL-TR-79-0001

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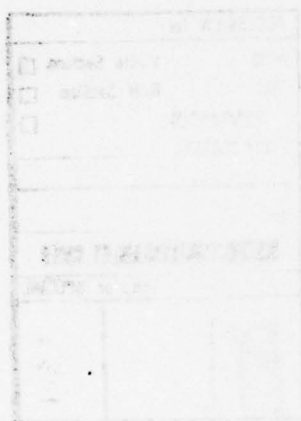
PREFACE

This report documents work done under Work Unit 2303-F2-07,
Pelletized Thermal Batteries, between 30 March and 15 October 1978.
The authors thank Donald Bush, Sandia Laboratories, for his help in
designing the single cell tester used in this work.

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INTRODUCTION

In previous studies we investigated the sodium tetrachloroaluminate (NaAlCl_4) electrolyte for use in thermally activated reserve cells (thermal cells). Lithium-aluminum alloys were used as anodes and MoCl_5 and CuCl_2 were used as cathode materials (1,2). A cathode screening study of 40 inorganic compounds indicated that FeCl_3 might exhibit performance superior to either MoCl_5 or CuCl_2 over a wider temperature range (3).

The electrochemistry of iron in equimolar AlCl_3 - NaCl was studied by Boxall *et al.* (4), who reported the Fe(III)/Fe(II) system to be reversible. The E° value for the Fe(III)/Fe(II) couple was $1.44 \pm 0.03\text{V}$ and for the Fe(II)/Fe(0) couple, $0.63 \pm 0.03\text{V}$. They also reported FeCl_3 to be quite soluble and FeCl_2 to be very insoluble in NaAlCl_4 melt.

The purpose of the present study was to investigate the discharge characteristics of the LiAl/FeCl_3 couple in the NaAlCl_4 electrolyte and to compare the performance of FeCl_3 cells to MoCl_5 and CuCl_2 cells.

The first task was to optimize the anode, separator, and catholyte compositions. Single cell tests of the optimum cell configuration were made at current densities of 15, 50, and 100 mA/cm^2 and at temperatures of 165, 175, 200, 225, and 250°C . Cell tests were also made at initial cell pressures from 560 to 7200 kg/m^2 .

EXPERIMENTAL

Electrolyte preparation, pellet fabrication, and single cell discharge experiments were conducted in an argon-filled glove box (Vacuum/

Atmospheres Co., Model HE 243-2 Dri-Lab/HE 193-2 Dri-Train) according to procedures given previously (1).

Iron(III) chloride (99.999%) was obtained in powdered form from Atomergic Chemetals Corp. and was used as received. The FeCl_3 particle size was roughly uniform (70 - 100 ASTM mesh) as determined using U.S.A. Standard Testing Sieves (W. S. Tyler, Inc.). Graphite (Grade 38 powder) was obtained from Fisher Scientific Co. and used as received.

Molybdenum foil (t3N) 0.25 mm thick was obtained from Alfa-Ventron, Inc. and used as current collectors. The foil was cut into 2.9 cm diameter circles with tab. The current collectors were burnished with 400 grit wet and dry sandpaper, washed with methanol and distilled water, and dried with acetone. The collectors were again lightly burnished immediately before use.

The single cell platen press used previously (1) was modified to incorporate a force transducer (Sensotec Inc., Model 20). The lower platen (see Fig. 1) was attached to a moveable base which rested on the force transducer. The upper platen was attached to a moveable piston. After the desired weight was placed on the piston, it was locked in position, giving a known initial stack pressure. The actual pressure encountered during heat up and discharge of the cell would then vary with time and extent of discharge. The pressure was monitored by an SCA 7-DI-3 amplifier (Sensotec Inc.), the output of which went to an HP 7100B recorder (Hewlett Packard, Inc.) through a digital multimeter (Honeywell Model 333).

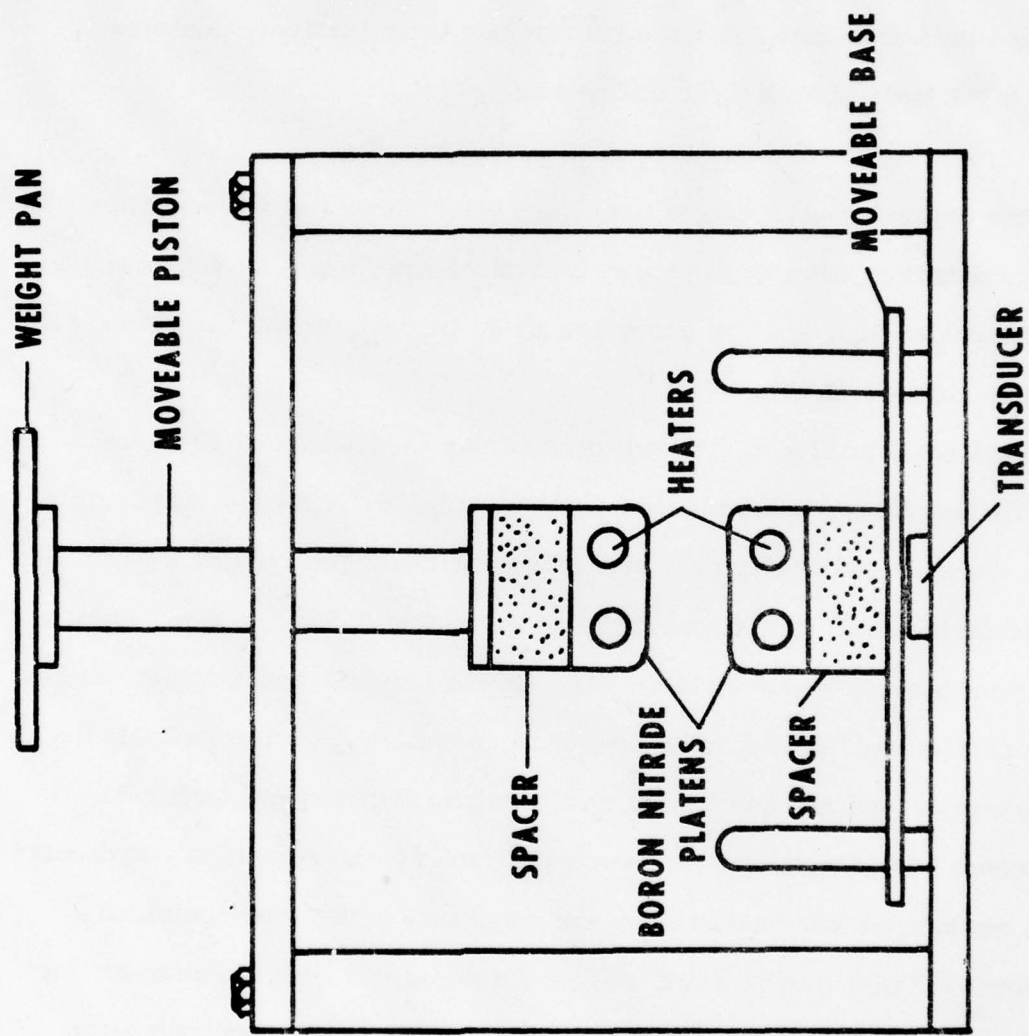


Figure 1. Simplified diagram of single cell tester.

The single cells were placed between the platens, the desired pressure applied, the platens heated to the desired temperature, and cell discharge initiated when the cell voltage stabilized. Constant current was maintained by a PAR Model 173 potentiostat/galvanostat (Princeton Applied Research Corp.) and the current was quantitatively measured with a PAR Model 179 digital coulometer.

RESULTS AND DISCUSSION

The criterion used to evaluate cell performance was the delivered energy density. Energy density calculations were based on the total mass of the pellet (1), and for discharge to 80 percent of initial closed circuit voltage (ICCV).

Cell optimization tests were carried out at 200° C and 15 mA/cm².

The weight of each component was optimized individually starting from the optimized MoCl₅ cell configuration (1). The results of the optimization study are summarized in Tables I - VI and the final optimum configuration is given in Table VII. The individual optimization studies were carried out in the order presented in Tables I - VI. Implicit in this process was the assumption that the configuration of each cell component was independent of the composition of the remaining components. This assumption was supported by the observation that the optimized weights of LiAl, graphite and EBM in the catholyte were the same as for the optimized MoCl₅ cell configuration. Still, the assumption may or may not be wholly valid, and certainly would need to be more carefully considered in any battery development study.

Electrolyte-binder mixture (EBM*) was added to the LiAl to facilitate fabrication of the anode layer. The desire was to add as little EBM as possible in order not to adversely effect the delivered energy density, yet still increase the ease of fabrication. As seen in Table VI, there was no significant difference in performance for added EBM weights from about 0.11g to about 0.14g. The value 0.12g was chosen to best facilitate anode fabrication without degrading cell performance.

The composition of the electrolyte and the amount of binder present in the electrolyte were not variables in the optimization process and therefore not necessarily the best values. They were, however, identical to those used in our previous studies.

*EBM 90 w/o electrolyte (49.85 m/o AlCl_3 , 50.15 m/o NaCl) and 10 w/o SiO_2 binder

TABLE I. Optimization of FeCl_3 weight^a.

<u>FeCl_3 weight (g)</u>	<u>Energy Density^b (Wh/kg)</u>
1.402	31.2
1.458	33.0
1.512	35.3
1.559	37.6
1.608	28.1

a. Cell configuration:

Anode	0.27g LiAl
Separator	0.78g EBM
Catholyte	<div style="display: inline-block; vertical-align: middle;"> <div style="font-size: 3em; vertical-align: middle; margin-right: 5px;">{</div> <div> 0.64g EBM FeCl_3 as indicated 0.23g graphite </div> </div>

b. To 80% ICCV.

TABLE II. Optimization of separator weight^a.

<u>Separator Weight (g)</u>	<u>Energy Density^b (Wh/kg)</u>
0.779	37.6
0.852	39.3
0.921	46.2
0.990	50.6
1.060	44.2

a. Cell configuration:

Anode	0.27g LiAl
Separator	EBM as indicated
Catholyte	<div style="display: inline-block; vertical-align: middle;"> <div style="font-size: 3em; vertical-align: middle; margin-right: 5px;">{</div> <div> 0.64g EBM 1.56g FeCl₃ 0.23g graphite </div> </div>

b. To 80% ICCV

TABLE III. Optimization of LiAl Weight^a.

<u>LiAl Weight (g)</u>	<u>Energy Density^b (Wh/kg)</u>
0.240	35.5
0.270	50.6
0.298	48.3
0.302	32.4

a. Cell configuration:

Anode	LiAl as indicated
Separator	0.99 g EBM
Catholyte	<div style="display: inline-block; vertical-align: middle;"> <div style="font-size: 2em; vertical-align: middle;">{</div> <div style="display: inline-block; vertical-align: middle;"> 0.64g EBM 1.56g FeCl₃ 0.23g graphite </div> </div>

b. To 80% ICCV.

TABLE IV. Optimization of graphite weight in catholyte^a.

<u>Graphite Weight (g)</u>	<u>Energy Density^b (Wh/kg)</u>
0.200	45.1
0.229	50.6
0.256	43.9

a. Cell configuration:

Anode	0.27 g LiAl
Separator	0.99 g EEM
Catholyte	<div style="display: inline-block; vertical-align: middle;"> <div style="font-size: 3em; vertical-align: middle; margin-right: 5px;">{</div> <div> 0.64 g EEM 1.56 g FeCl₃ graphite as indicated </div> </div>

b. To 80% ICCV

TABLE V. Optimization of EBM in catholyte^a.

<u>EBM Weight (g)</u>	<u>Energy Density^b (Wh/kg)</u>
0.583	41.9
0.638	50.6
0.700	47.7

a. Cell configuration:

Anode	0.27g LiAl
Separator	0.99g EBM
Catholyte	<div style="display: inline-block; vertical-align: middle;"> <div style="font-size: 2em; vertical-align: middle; margin-right: 5px;">{</div> <div> EBM as indicated 1.56g FeCl₃ 0.23g graphite </div> </div>

b. To 80% ICCV.

TABLE VI. Weight of EBM added to anode^a.

<u>EBM Weight (g)</u>	<u>Energy Density^b (Wh/kg)</u>
0.110	46.1
0.113	46.4
0.132	46.0
0.146	47.3

a. Cell configuration:

Anode	{ 0.27g LiAl EBM as indicated
Separator	0.99g EBM
Catholyte	{ 0.64g EBM 1.56g FeCl ₃ 0.23g graphite

b. To 80% ICCV.

TABLE VII. Optimum cell configuration

Anode	{ 0.27 g LiAl (60 a/o Li) 0.12 g EBM
Separator	0.99 g EBM
Catholyte	{ 0.64 g EBM 1.56 g FeCl ₃ 0.23 g graphite

Cell Characteristics

The open circuit voltage (OCV) of the FeCl₃ cells was 2.36 to 2.38V at temperatures of 175°C and above. At 165°C, the OCV was 2.32V. The internal resistance was determined by measuring initial closed circuit voltage under various loads on a series of cells. Fig. 2 shows that cell resistances at different current densities vary between about 0.73 Ω to about 0.36 Ω at 175°C. The internal cell resistance then is of the order of 0.5 Ω . This value was relatively constant over the temperature range studied.

A yellow color was observed throughout the separator layer after every cell was discharged, indicating that the cathode reaction was a single electron reduction of Fe(III) to Fe(II).

The cell resistance as a function of extent of discharge also supports the Fe(III) to Fe(II) cell reaction. The cell resistance as a function of extent of discharge was determined using the method described in a previous study (5), and the results are graphed in Fig. 3. The rapidly increasing resistance at about 70% of discharge indicates a single insoluble product.

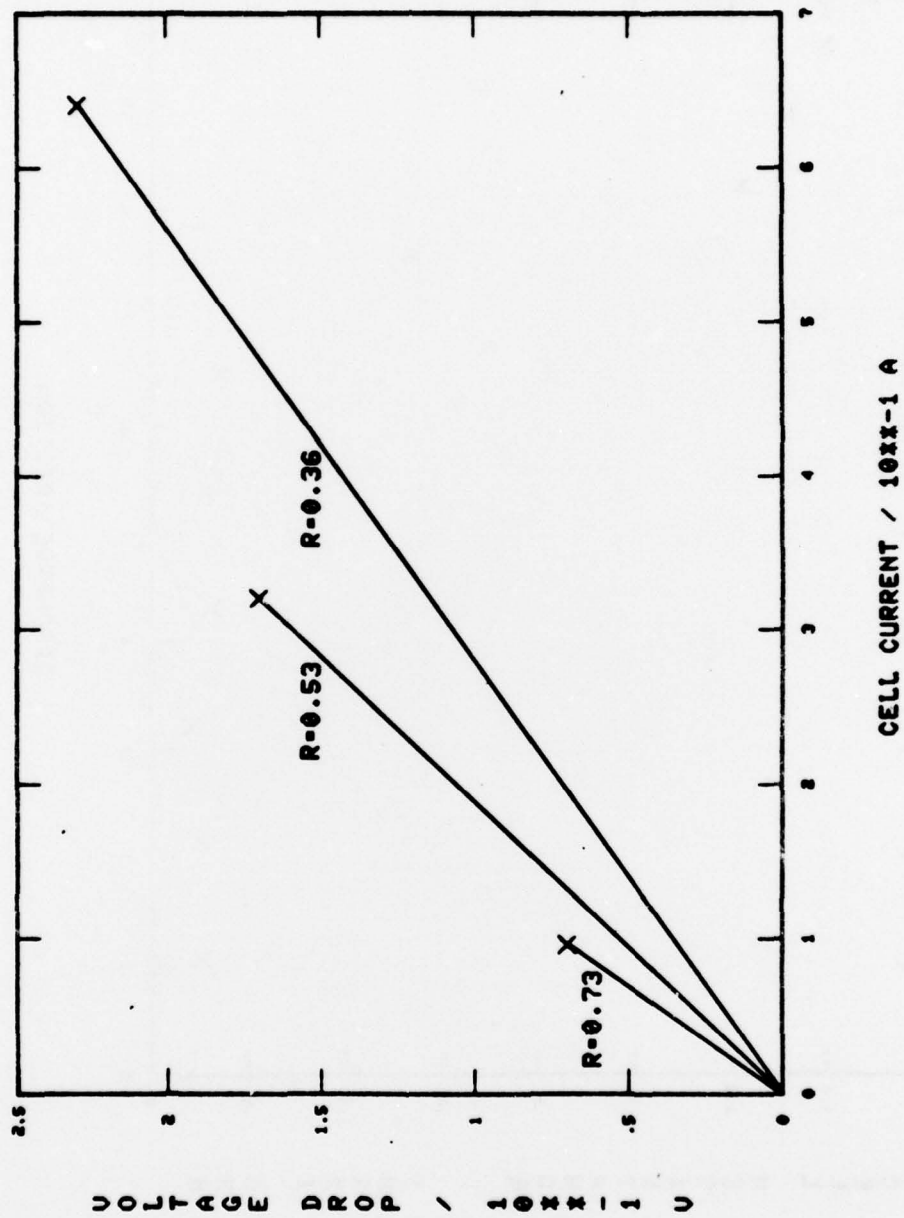


Figure 2. Internal resistance of FeCl_3 cells (175 C and 15 mA/cm^2)

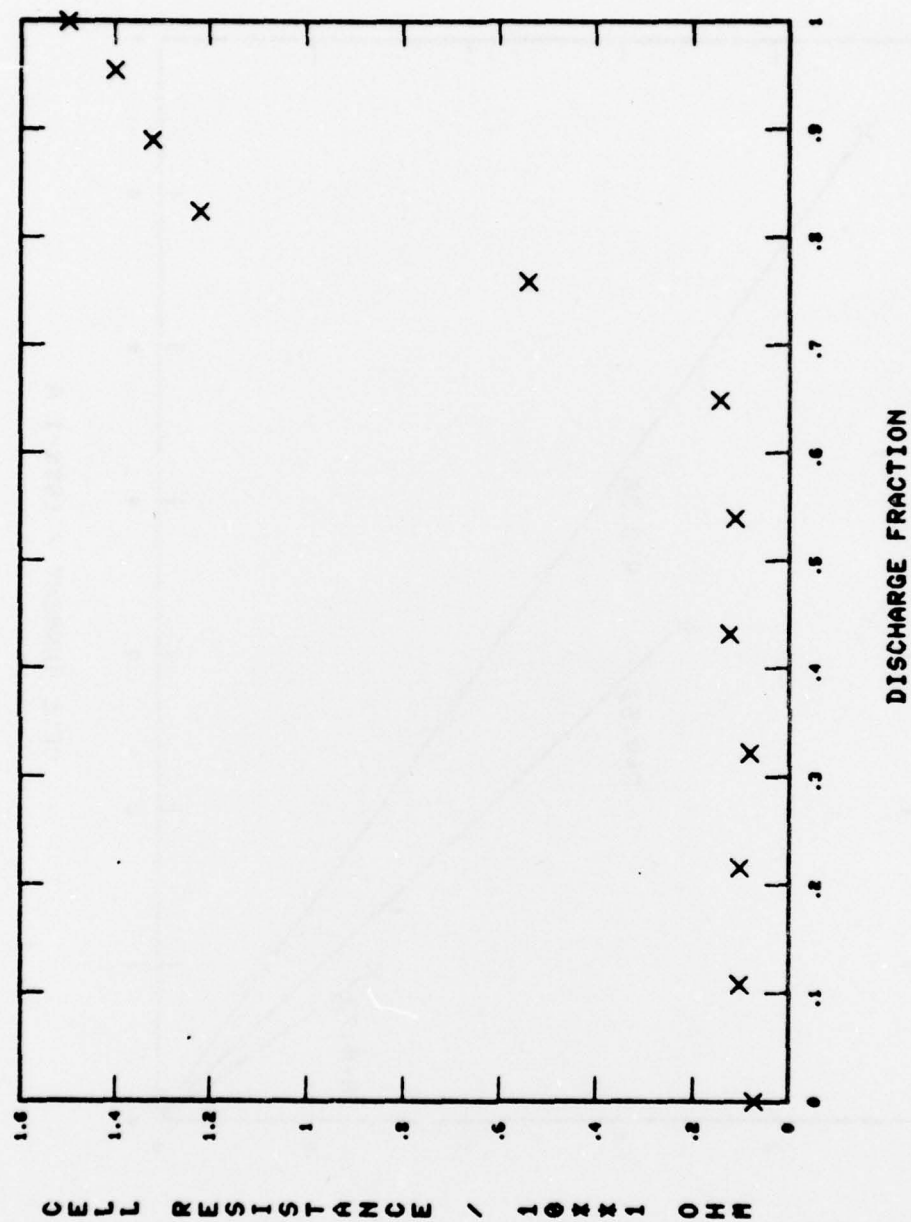


Figure 3. Cell resistance of FeCl3 cells as a function of extent of discharge (175 C and 15 mA/cm²).

Cell Discharge Behavior

Cell discharge experiments were performed to study the effects of stack pressure, temperature, and discharge rates on cell performance. Initial stack pressure was varied from 560 to 7200 kg/m², the temperature range was 165 to 250°C, and discharge rates were 15, 50, and 100 mA/cm².

Our results of varying stack pressure using the NaAlCl₄ electrolyte (Fig. 4) were similar to the results obtained by Bush with the LiCl-KCl electrolyte (6). At initial pressures greater than about 1400 kg/m², there appeared to be little dependence upon pressure of the energy densities obtained. Pressure varied in a roughly reproducible manner during cell discharge for all combinations of temperature, current density, and initial stack pressure (above ca. 1400 kg/m²). A maximum of 3 to 4 times the initial stack pressure was reached early in the discharge, followed by a relative minimum at about 80% of ICCV, then another maximum occurred, and finally the pressure decayed to approximately the initial value at 0.0V. We have made no attempt to relate the pressure changes to specific cell processes; however, there appeared to be a noticeable correlation between the pressure/discharge profile of a given cell and whether or not that cell delivered its full expected energy.

Typical discharge curves for various temperatures and discharge rates are shown in Fig. 5 and Fig. 6 respectively, and the corresponding energy densities are given in Table VIII.

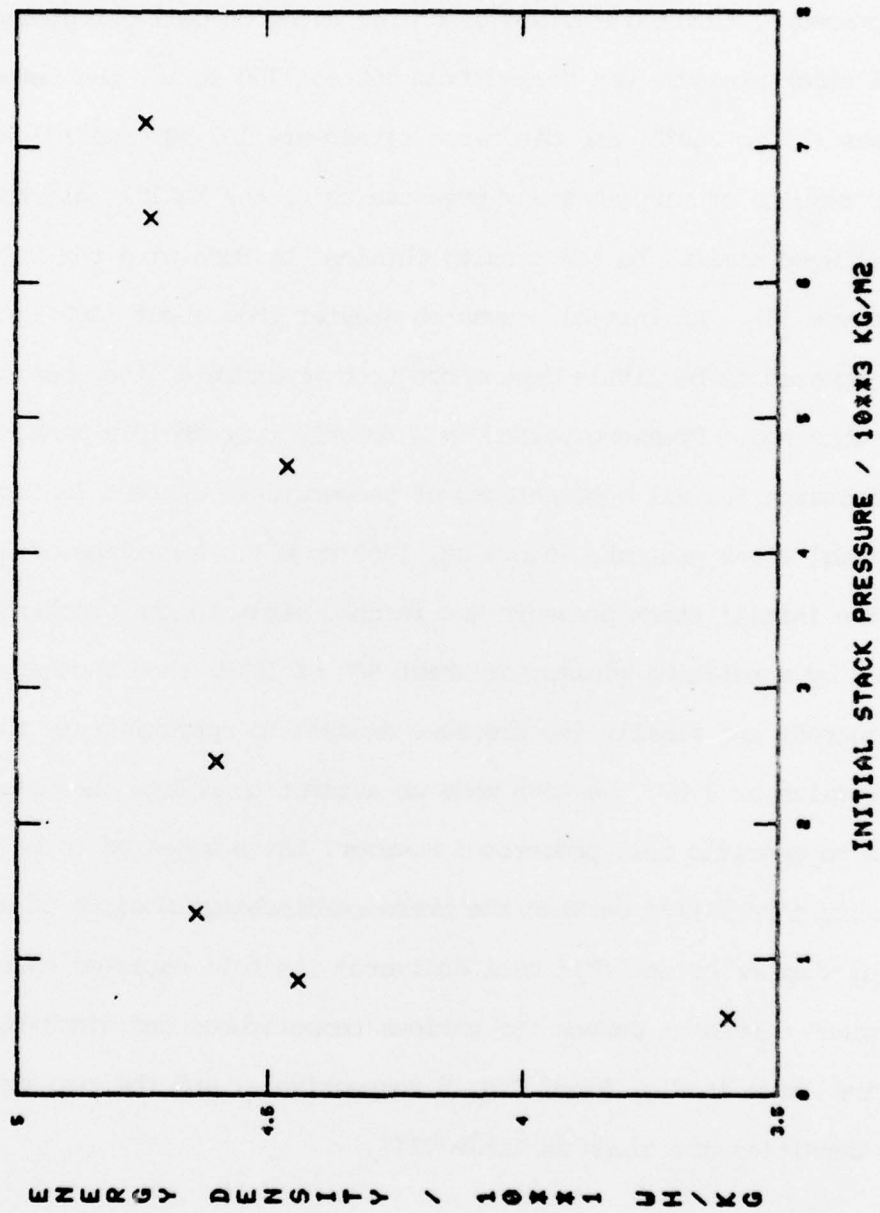


Figure 4. Dependence of energy density on initial stack pressure.

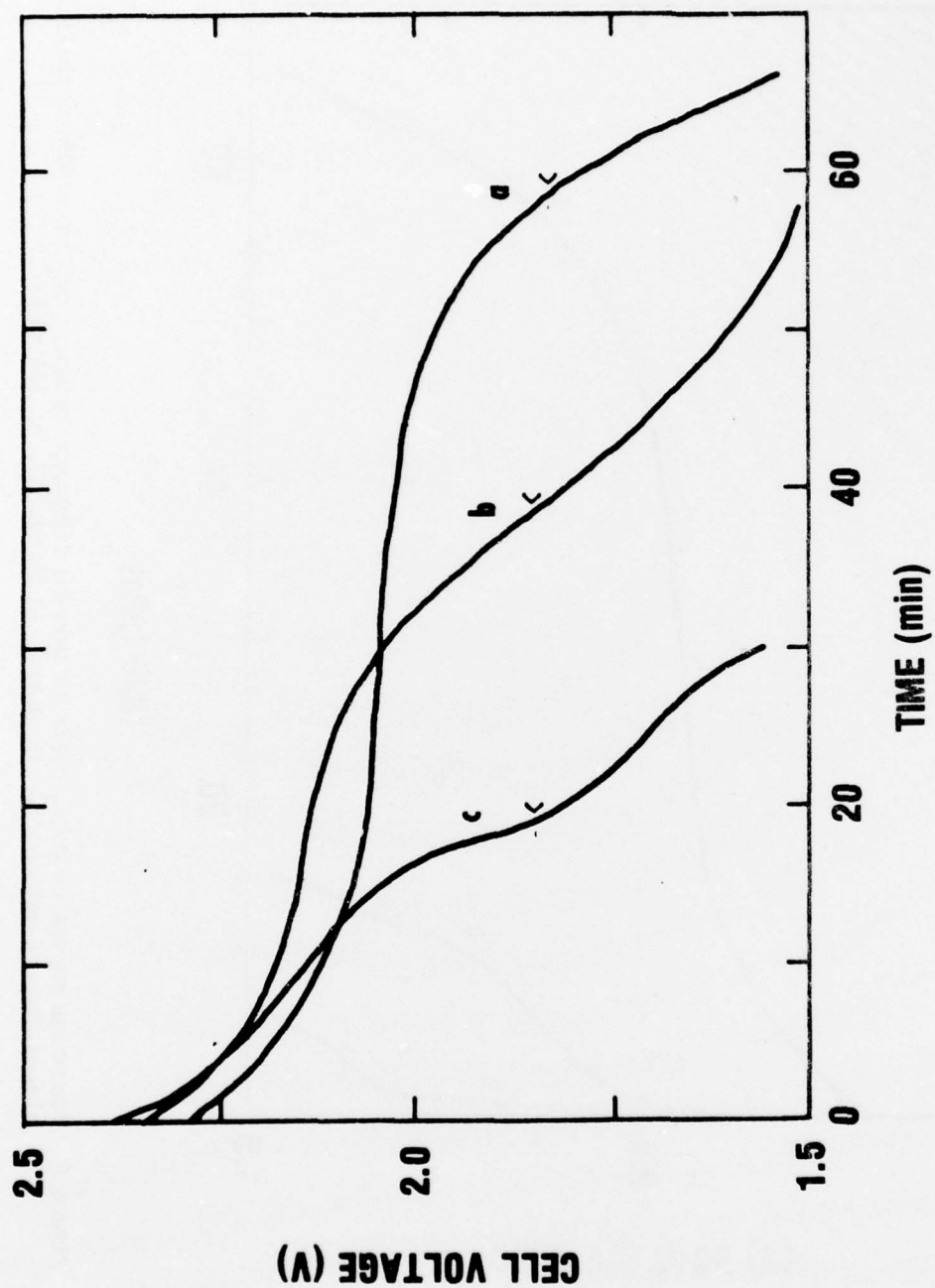


Figure 5. Discharge curves of FeCl_3 cells at various temperatures (15 mA/cm^2). < indicates 80% of initial closed circuit voltage; a) 175°C , b) 225°C , c) 250°C .

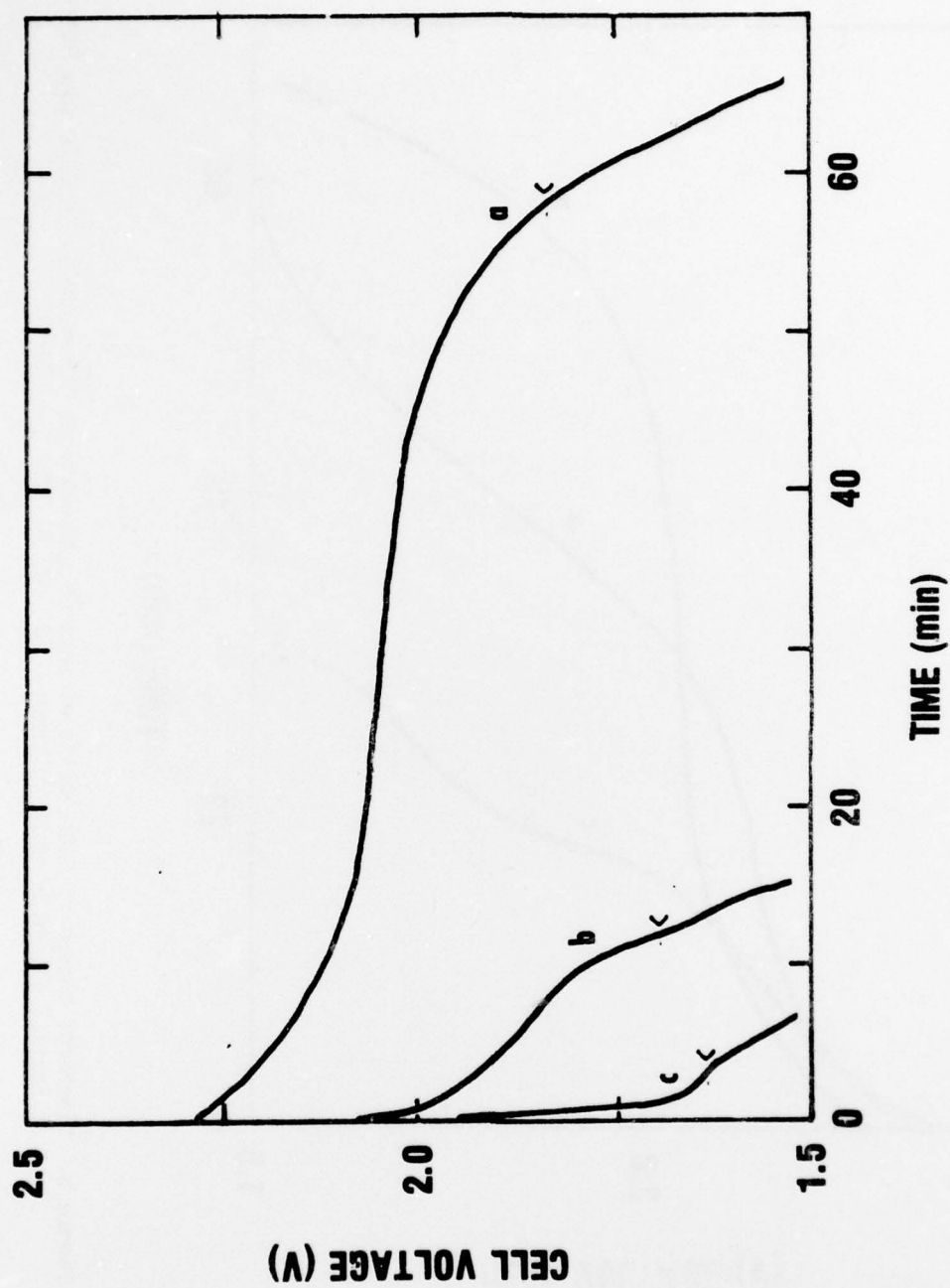


Figure 6. Discharge curves of FeCl₃ cells at various discharge rates. < indicates 80% of initial closed circuit voltage; a) 15 mA/cm², b) 50 mA/cm², c) 100 mA/cm².

TABLE VIII. Energy densities obtained for FeCl_3 cells at different current densities and temperatures.

Current Density (mA/cm ²)	Energy Density ^a (Wh/kg)				
	165°C	175°C	200°C	225°C	250°C
15	42.1	50.6	46.4	38.1	17.2
50	36.1	31.7	31.9	44.4	34.9
100	12.1	14.9	32.2	36.7	33.4

a. 80% ICCV

Cell lifetimes are considerably shortened with increased temperature and increased current density; however, delivered energy density remained high in most cases. Those conditions for which cell performance was significantly reduced were the low temperature-high current density and the high temperature-low current density conditions, as seen in Table VIII, and in Fig. 7. These performance losses may be due to diffusional limitations or to the increased cell resistance as Fe(II) formation progresses during cell discharge. The optimum operating temperatures were 200-250°C where, as seen in Fig. 7, cell performance was relatively stable for the current density range studied.

Comparison of FeCl_3 cells with MoCl_5 and CuCl_2 cells.

The parameter used to compare the different types of cells was the percent change in performance defined by the following relation:

$$\% \text{ Change} = \frac{\text{Maximum Energy Density} - \text{Minimum Energy Density}}{\text{Maximum Energy Density}} \times 100$$

This parameter relates the relative performance of cells at constant current density with varying temperature, and at constant temperature

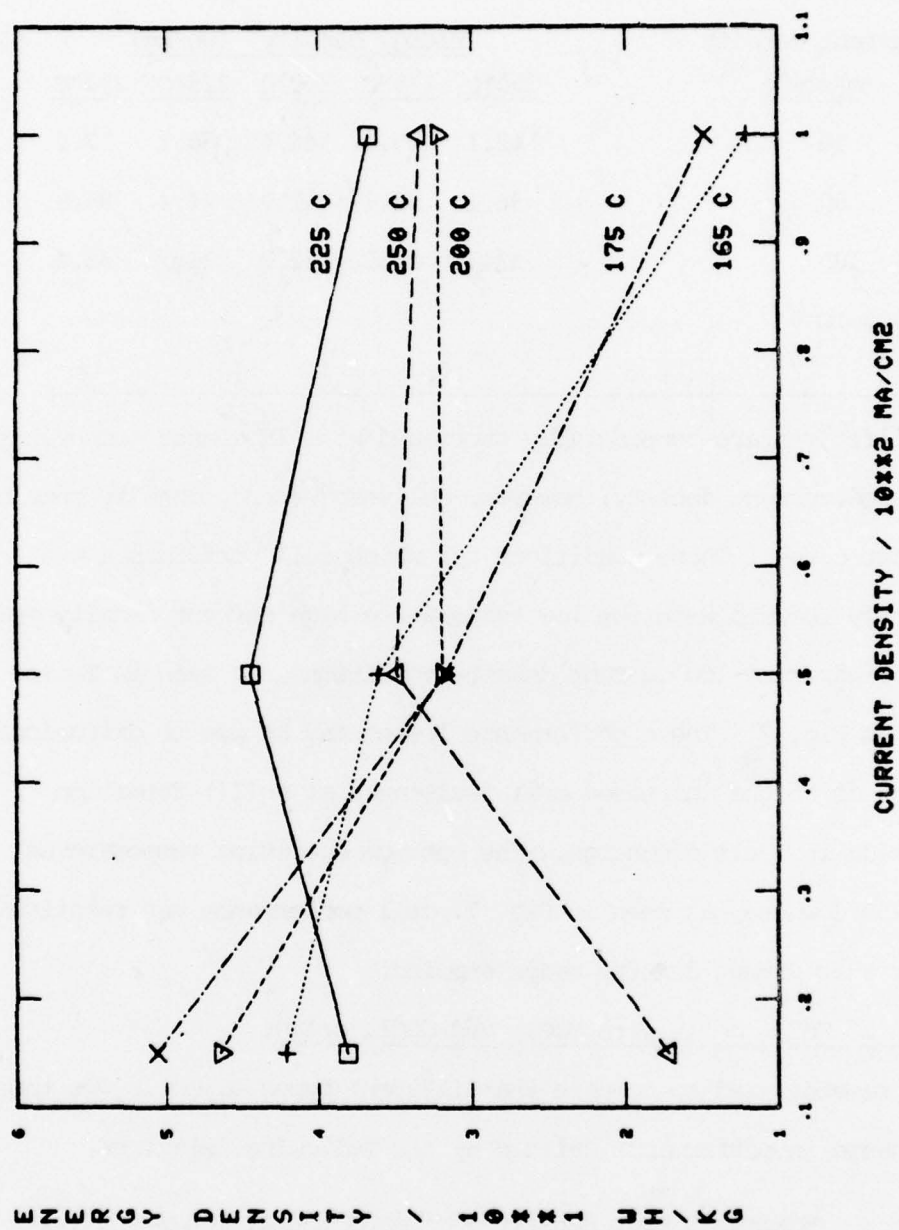


Figure 7. Energy densities of FeCl_3 cells at various temperatures as a function of current density.

with varying current density. The smaller the value of the parameter, the more tolerant the cell is toward the changing operating conditions. Percent changes for cells discharged at constant current densities are tabulated in Table IX.

TABLE IX. Comparison of FeCl_3 , MoCl_5 , and CuCl_2 cells over the temperature range 165°C to 250°C at constant current densities.

Current Density (mA/cm ²)	Cell Type	Energy Density ^a (Wh/kg)					Percent Change ^b
		165°C	175°C	200°C	225°C	250°C	
15	CuCl_2	-	19.8	25.3	12.3	4.8	81.0
	MoCl_5	29.7	36.3	26.4	20.4	15.0	58.7
	FeCl_3	42.1	50.6	46.4	38.1	17.2	66.0
50	MoCl_5	12.3	17.6	27.7	24.4	21.8	55.6
	FeCl_3	36.1	31.7	31.9	44.4	34.9	28.6
100	MoCl_5	-	-	36.1	14.3	21.1	60.4
	FeCl_3	12.1	14.9	32.2	36.7	33.4	67.0
							(12.3) ^c

a. To 80% ICCV

b. Percent change is defined as

$$\frac{\text{Maximum Energy Density} - \text{Minimum Energy Density}}{\text{Maximum Energy Density}} \times 100$$

c. Value in parentheses is for the same temperature range as MoCl_5 data.

The FeCl_3 cell performance was substantially better than MoCl_5 and CuCl_2 cells at 15 mA/cm² discharge rate at each temperature studied, and the percent change in performance for FeCl_3 cells was comparable to or

better than MoCl_5 and CuCl_2 cells at that discharge rate. At higher discharge rates, FeCl_3 cells were definitely superior to MoCl_5 cells. Data for CuCl_2 cells at temperatures other than 175°C were not available at 50 and 100 mA/cm^2 , therefore, no valid comparison could be made between CuCl_2 and FeCl_3 cells at the higher discharge rates.

Table X compares the percent change in performance for FeCl_3 and MoCl_5 cells under varying discharge rates at each temperature studied.

TABLE X. Percent change in performance for FeCl_3 and MoCl_5 cells for the current density range 15 to 100 mA/cm^2

<u>Temperature ($^\circ\text{C}$)</u>	<u>Percent Change</u>	
	<u>MoCl_5</u>	<u>FeCl_3</u>
165	55.6 ^a	71.3 (14.3) ^b
175	51.6 ^a	70.6 (37.4) ^b
200	26.9	31.3
225	41.4	17.3
250	31.2	50.7

- a. MoCl_5 data is over the current density range 15 to 50 mA/cm^2 .
 b. Values in parentheses is for the same current density ranges as MoCl_5 data.

At temperatures below 250°C , FeCl_3 cell performance is superior to MoCl_5 cell performance for the same current density ranges.

CONCLUSIONS

Iron(III) chloride was an excellent cathode material for thermal cells using a NaAlCl_4 electrolyte. The optimum operating temperature of

FeCl_3 cells based on percent change in performance was 200–250°C where the delivered energy density (to 80% ICCV) varied from 31.9 to 46.4 Wh/kg over the full current density range studied. The best energy density (50.6 Wh/kg) was obtained at 175°C and 15 mA/cm².

Iron(III) chloride cell performance was compared to MoCl_5 cell performance. At a given current density, FeCl_3 cells suffered less performance loss over the temperature range 165°C to 250°C than cells containing MoCl_5 . At a given temperature, FeCl_3 cell performance was usually superior to MoCl_5 cells when compared over the same current density range.

Several areas need to be investigated further. The compaction pressures used during cell fabrication need to be studied to determine if cell performance is effected. The effects, if any, of FeCl_3 purity need to be studied. The FeCl_3 used in this study was 99.999 percent pure and cost \$175 for 100 g. However, the same supplier offers FeCl_3 that is 96% pure for \$12.50 for 250 g. This was the first study done in this laboratory in which the pressure changes could be observed during cell discharge. The results obtained indicate that further study and data are needed in this area.

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